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INVESTIGATION OF ADVANCED PROTECTIVE
AND ANTIREFLECTION COATINGS FOR
HALIDE OPTICS

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Rockwell International Corporation

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n-butyl and isopropyl alcohols and a Syntron Polisher, both with and without polishing powders. Surface removal rates of about 600 Å/minute were established.

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CONTENTS

	<u>Page</u>
1.0 Introduction	1
1.1 Program Objective	1
1.2 Work Statement	1
2.0 Progress By Task	2
2.1 Monomer Selection	2
2.2 Polymer Deposition Equipment	3
2.3 Polymer Deposition	5
2.4 Potassium Chloride Polishing	6
3.0 Summary	11
References	12

ILLUSTRATIONS

<u>Figure</u>	<u>Page</u>
1. Equipment Used for Both Electrodeless Glow Discharge and Ultraviolet Photolytic Polymerizations	4
2. Scanning Electron Micrographs of Harshaw Potassium Chloride Windows Before Polishing Experiments	8
3. Removal of KCl by Non-Abrasion Polishing in Isopropyl Alcohol Using a Syntron Polisher	10

TABLES

<u>Table</u>	<u>Page</u>
I. Monomers Selected for Evaluation	3
II. Results of Ultraviolet Deposition of Dichlorodifluoromethane and Bromotrifluoroethylene	5
III. Solubilities of KCl in Several Alcohols at 25°C	7

1. INTRODUCTION

1.1 PROGRAM OBJECTIVE

The objective of this program is to conduct an integrated evaluation of techniques for surface finishing and protective coating of alkali halide substrates for use in laser systems operating in the 10.6 and 3 to 5 μm regions. Potassium chloride has been selected as the alkali halide substrate to be studied.

Surfaces lapped and polished using conventional abrasive techniques will be compared with those resulting from chemical lapping, chemical lapping followed by glow discharge cleaning, and sputter etching. Thin organic polymeric coatings prepared by electrodeless glow discharge polymerization and ultraviolet irradiation will be evaluated for their ability to provide environmental protection and the required optical properties.

A study will also be made of possible antireflective coating designs, the selection and deposition of appropriate organic materials, and measurement of the antireflective properties of these films.

1.2 WORK STATEMENT

Conduct an integrated research program on the preparation and evaluation of organic polymeric infrared optical coatings for alkali halide substrates. Surface finishing will be done using chemical-mechanical techniques and ion polishing. These surfaces will be characterized by visual and microscopic examination with scanning electron microscopy, as well as measurements of scattering, reflectance, transmittance and absorptance. Surface coatings will be prepared by ultraviolet or radio frequency glow discharge polymerization or copolymerization of the appropriate monomers. In addition to the previously mentioned techniques, the coatings will be evaluated by absorption calorimetry, interferometry, X-ray or electron diffraction, and tests for mechanical adhesion. Window surfaces will be subjected to high power laser pulses before and after coating in order to establish damage thresholds for the surface and the coatings. Indices of refraction will be precisely measured for all the materials selected, possible antireflective coating designs will be configured, and the appropriate materials will be deposited and the antireflective properties will be measured.

Samples (Polymer Coated Alkali Halide Optics) developed under the contract will be delivered, at the request of the Contracting Officer, and shall not exceed six samples per quarter.

2. PROGRESS BY TASK

2.1 MONOMER SELECTION

Thin organic polymeric coatings have been prepared by glow discharge polymerization (electrode or electrodeless), electron bombardment, sputtering, and irradiation with ultraviolet light. The resultant coatings are characterized as amorphous, thermally stable materials with good adhesion and low porosity. If the above properties can be combined with low optical absorption, low water vapor transmission and abrasion resistance, the resultant coating should be suitable for the protection of alkali halide windows for infrared laser systems.

A review of the infrared spectra of thin film polymers resulting from the glow discharge or ultraviolet polymerization of methane⁽¹⁾, ethane⁽¹⁾, ethylene⁽²⁾, propane⁽¹⁾, butane⁽¹⁾, butadiene⁽²⁾, benzene⁽²⁾, styrene^(2 and 3), naphthalene⁽²⁾ and silicone oil⁽⁴⁾ (electron bombardment) showed the presence of absorption bands in the 10 to 11 μm region due to $\text{RHC} = \text{CHR}$ and $\text{RCH} = \text{CH}_2$ groups in the polymers and bands in the 3 to 4.5 μm region due to $-\text{CH}_2-$, $-\text{CH}_3$, R_3CH , $-\text{C} = \text{C}-$, and $-\text{C} \equiv \text{C}-$ or $-\text{C} = \text{C} = \text{C}-$ groups. These polymers also contained trapped free radicals which, on exposure to air, reacted with oxygen and water vapor giving rise to carbonyl and hydroxyl absorptions. The polymer from silicone oil also showed a broad absorption band at 10.5 μm due to $\text{Si}-\text{O}-\text{Si}$ stretching. Therefore, polymers prepared from hydrocarbon-type monomers offer little potential for providing nonabsorbing windows in the 3 to 5 and 10.6 μm regions.

The halocarbons on the other hand appear to offer excellent potential as protective coatings for potassium chloride optics. In preliminary work prior to this contract, ultraviolet polymerized films of polyhexachlorobutadiene on sodium chloride crystals were subjected to high power 10.6 μm radiation. The films exhibited the ability to survive extremely high power densities with breakdown occurring at 2 MW/cm^2 . These films, as confirmed by Wright⁽⁵⁾, are essentially transparent over the 2 to 15 μm range.

Prior work, also at Autonetics Division of Rockwell International⁽⁶⁾, on the glow discharge polymerization of 1,1-difluoroethylene, 1,1-difluoroethane, 1,1-difluoro-1-chloroethane, trifluoroethylene, tetrafluoroethylene, and fluoroform, also show high potential for the halocarbon polymers as protective coatings for the halide optics. All of the polymers showed the presence of oxygen as evidenced by the carbonyl absorption at about 5.9 μm . The oxygen could have been introduced during the polymerization or upon exposure of the films to atmospheric oxygen. The system was evacuated to only 10^{-3} Torr prior to the polymerization and residual oxygen, therefore, remained in the system. All of the polymers prepared from monomers containing hydrogen with the exception of fluoroform, showed the presence of hydroxyl absorptions at about 3.0 μm and C-H absorptions at about 3.4 μm . The polymers from tetrafluoroethylene and fluoroform showed no absorption bands in the 3 to 5 μm region. None of the polymers exhibited distinguishable absorption bands in the 10 to 15 μm region.

Fifteen monomers listed in Table I containing varying percentages of fluorine, chlorine, and bromine combined with saturated, unsaturated (single and conjugated double bonds), and cyclic structures were selected for evaluation. The selection system was based on obtaining the maximum number of bond types and structures

TABLE I. Monomers Selected for Evaluation

<u>Monomer</u>	<u>Chemical Structure</u>
Fluororform	HCF_3
Bromotrifluoromethane	BrCF_3
Chlorotrifluoromethane	ClCF_3
Dichlorodifluoromethane	Cl_2CF_2
Trichlorofluoromethane	Cl_3CF
Tetrachloroethylene	$\text{Cl}_2\text{C} = \text{CCl}_2$
Tetrafluoroethylene	$\text{F}_2\text{C} = \text{CF}_2$
Chlorotrifluoroethylene	$\text{ClFC} = \text{CF}_2$
Bromotrifluoroethylene	$\text{BrFC} = \text{CF}_2$
Chloropentafluoroethane	$\text{ClF}_2\text{C} - \text{CF}_3$
1,2-Dichlorotetrafluoroethane	$\text{ClF}_2\text{C} - \text{CF}_2\text{Cl}$
1,2-Dibromotetrafluoroethane	$\text{BrF}_2\text{C} - \text{CF}_2\text{Br}$
1,1,2-Trichlorotrifluoroethane	$\text{Cl}_2\text{FC} - \text{CF}_2\text{Cl}$
Hexafluorocyclobutane	$\text{F}_2\text{C} - \text{CF}_2 - \text{CF}_2 - \text{CF}_2$
Hexachlorobutadiene	$\text{Cl}_2\text{C} = \text{CCl} - \text{CCl} = \text{CCl}_2$

using a minimum number of monomers. This should aid in resolving the compositional and structural relationships of the resultant polymers with their chemical, mechanical, and optical properties. It will also aid in the selection of monomer blends to be used for altering these properties particularly in the development of antireflective coatings.

2.2 POLYMER DEPOSITION EQUIPMENT

A photo of the equipment used in the polymerizations is shown in Figure 1.

The glow discharge chamber is contained in the bell jar on the left side of the picture and the photolysis chamber is the glass cross on the right. The photolysis chamber is mounted on a four inch oil diffusion pump separated from the chamber by a two inch valve and liquid nitrogen trap. The glow discharge chamber is connected to the photolysis chamber through one inch flexible stainless steel tubing and valving to permit independent operation of the two systems. The discharge coil is coupled

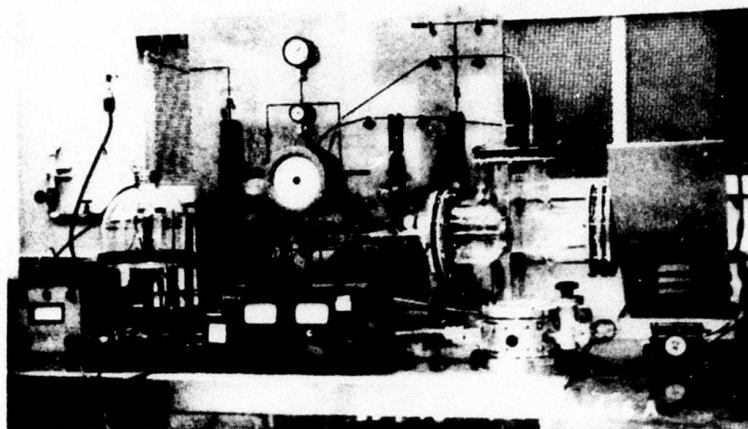


Figure 1. Equipment Used for Both Electrodeless Glow Discharge and Ultraviolet Photolytic Polymerizations

to an R.D. Mathis Co., Model SP310, rf generator operating at 13.56 MHz and delivering continuously variable output power from 0 to 1000 watts. The ultraviolet source is a Hanovia 929B - 9U, 2500 watt mercury-xenon lamp mounted in an Electro Powerpaks Corporation, Model 371, housing. Vacuums in the 10^{-6} torr range can be achieved with this system. To avoid polymer contamination all of the pumps contain liquid nitrogen traps and the vacuum system is made entirely of glass and metal parts with Viton O-rings as seals.

Preliminary experiments revealed that a glow discharge can also be achieved in the photolytic chamber by placing a discharge coil around the arm of the cross adjacent to the ultraviolet lamp housing. This should permit glow discharge cleaning of the potassium chloride substrate prior to the photolytic deposition. Deposition by a combination of glow discharge and ultraviolet photolysis is also possible. Thicker than normal coatings should be achievable by this technique using a low power glow discharge to control the amount of cross-linking in the polymer.

Several critical parts for the high vacuum system were not received until the end of this quarter and the lowest vacuum that could be achieved in the chambers until that time was 10^{-3} torr. This was adequate, however, to permit coupling and tuning of the rf generator to the coil on the glow discharge and photolytic chambers. Several deposition runs were made using the photolytic chamber as described below. Both chambers are expected to be fully operational during the second week of the third quarter.

2.3 POLYMER DEPOSITION

As discussed above, the maximum vacuum which could be achieved in the chambers prior to receipt of several parts for the manifold was 10^{-3} mm Hg. The photolytic chamber was therefore evacuated to this pressure, backfilled with argon and re-evacuated several times to remove residual air. Two weighed aluminum substrates (18 x 50 x 0.1 mm) were used for each deposition. The gaseous monomers selected were dichlorodifluoromethane (Cl_2CF_2) and bromotrifluoroethylene (BrC_2F_3). The reaction conditions and results are shown in Table II.

TABLE II. Results of Ultraviolet Deposition of Dichlorodifluoromethane and Bromotrifluoroethylene

Run	Monomer	Flow Rate cc/min	Pressure mm Hg	Substrate Temp., °C	Deposition Time, min.	Wt. Deposited mg/cm ²
1	Cl_2CF_2	9.6	0.22	18-36	90	0
2	BrC_2F_3	8.6	0.27	23-39	60	0
3	BrC_2F_3	8.6	7	22-33	60	-
		0	7-0.028	33-37	60	0.09

No polymer could be detected in run (1) using dichlorodifluoromethane by either weight gain of the aluminum substrates or by infrared analysis performed by placing the substrates against a KRS-5 crystal mounted in a Frustrated Multiple Internal Reflection (FMIR) attachment to a Perkin Elmer, Model 454, infrared spectrophotometer.

Bromotrifluoroethylene (BrC_2F_3) was selected for the second run since it contained a carbon-to-carbon double bond and weaker carbon-to-bromine bond, and should therefore be more readily polymerizable using ultraviolet light. The reaction conditions were almost identical to those used in run (1). Again no polymer was detectable.

The pressure was increased in run (3) by partially closing the valve to the vacuum pump. After one hour of exposure to the 2500 watt mercury-xenon lamp liquid condensate had formed on the substrates and on the walls of the vacuum chamber. The gas flow was stopped and the valve to the vacuum pump was fully opened. Ultraviolet irradiation was continued for an additional hour while the pressure slowly dropped to 0.028 torr and liquid condensate could no longer be detected on the walls of the vacuum chamber. A tacky colorless film weighing 0.09 mg/cm² resulted. If a density of 2.0 is assumed for the film, the film thickness is approximately 4.5×10^{-5} cm. FMIR analysis again showed no absorption bands other than those of the uncoated aluminum substrate. The film was readily soluble in acetone indicating a low molecular weight.

While these results are too limited to draw any conclusions, they indicate the importance of monomer pressure on the deposition rate. The reason for the failure of FMIR to show the presence of the polymer on the substrate in run (3) is unknown at this time. Thinner substrates will be used to obtain a better press fit against the KRS-5 crystal and if this fails, the crystal itself will be used as the substrate.

2.4 POTASSIUM CHLORIDE POLISHING

Experimental studies were initiated on the use of anhydrous alcohols as vehicles for soft abrasive polishing of potassium chloride laser windows. The purpose of the alcohol is to provide a protective ambient for the window discs as well as to provide a slowly reacting solution of very low equilibrium solubility for the potassium chloride. The higher alcohols (three carbons and up) offer an easily controlled solubility for KCl, with dissolution values decreasing up the series and with solubility in the primary alcohol less than in the corresponding secondary (see Table III). The combination of etching (by dissolution) together with controlled light abrasive action should provide as excellent a final polished surface as the analogous technique does for silicon wafers. In the absence of water, the techniques reported by Sakamoto and Kobayashi⁽⁷⁾ and others, should be improved. Initial tests will involve alcohols alone or with standard aluminum oxide polishing powders, but later work will concentrate on softer, more friable (i. e., more easily broken down to finer particle size) abrasives.

For these initial tests, a Syntron Polisher, Model LP01C, was used to provide gentle agitation during polishing. The bowl bottom was fitted with a glass plate finely lapped to an extremely flat condition. Over this was positioned a polishing cloth surface (i. e., micro cloth, nylon cloth or silk cloth). A polishing vehicle volume of 400 ml was used for each experimental run. The rate of surface removal was determined by weighing to 0.0001 gram using a semi-automatic, one-pan, Ohaus Model 1600 balance.

Potassium chloride windows, optically polished (5-10 fringe flatness, parallel to 10 minutes), had been obtained from Harshaw in the form of discs 25 mm in diameter and 5 mm thick. Later work will involve Harshaw laser quality discs flat to 1/10 wavelength, parallel to 30 seconds (see Figure 2d). These flat windows were inspected at 30X and 400X under a binocular microscope in order to characterize the surface. Microphotographs and scanning electron micrographs (SEM) (see Figures 2a, 2b, and 2c) were taken of selected areas in order to compare later polishing effects. After weighing the 5 gram discs to five significant figures, the windows were allowed to randomly rotate on a Syntron Polisher; after measured time intervals the discs were reweighed to determine thickness changes.

Butyl alcohol, alone or with a few percent of 0.05 micron size aluminum oxide (Linde B abrasive) appeared to produce minimal action with a micro cloth surface. Butyl alcohol, with a few percent of 9 micron size (Microgrit WCA No. 9) Al_2O_3 on a nylon cloth appeared to remove about 700 Å/minute at a low Syntron speed and a pressure of about 7 grams/cm². However, this coarse, hard material produced easily visible scratches on the potassium chloride surface. The damaged surface was further polished using butyl alcohol vehicle with the smaller size 1 micron Al_2O_3 (Microgrit GB 2000) polishing powder using the Syntron vibrator with a nylon cloth and the same 7 grams/cm² pressure. This combination appeared to remove surface material at a rate of about 500 Å/minute. The surface finish, although showing improvement, appeared to change slowly.

TABLE III. Solubilities of KCl in Several Alcohols at 25°C*

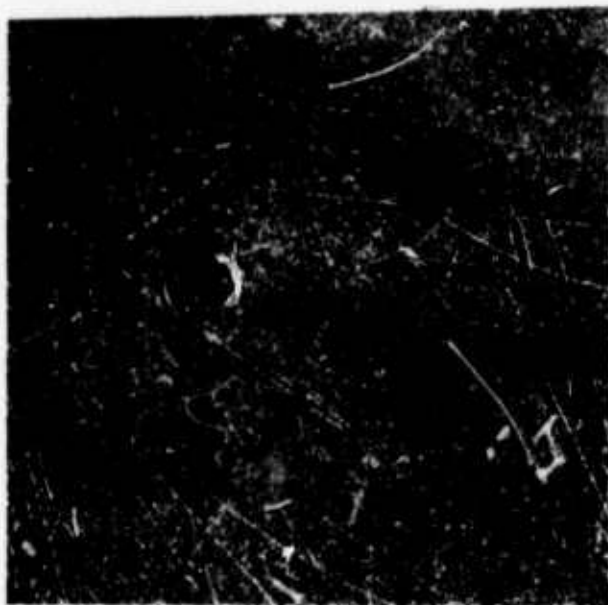
<u>Alcohol</u>	<u>Percent KCl by Weight</u>
Methyl Alcohol	0.53
Ethyl Alcohol	0.028
n-propyl Alcohol	0.0040
Isopropyl Alcohol	0.021
n-butyl Alcohol	0.0012
Isobutyl Alcohol	0.0049
Isopentyl Alcohol	0.0009

* (Interpolated from data given in "A Few Rules Concerning the Variations of the Solubility of the Chlorides of Alkali Metals in Alcohols," by V. Plyushev et al, *Nauk. Doklady Vysskei Shkoly, Khim. i Khim Tekhnol.* 1958, No. 2, p. 279-282)

A less expensive organic solvent and one with higher solubility for potassium chloride appeared appropriate from the above preliminary work. Isopropyl alcohol satisfies both requirements (see Table III), having a nearly twenty-fold higher solubility for potassium chloride over n-butyl alcohol. This higher equilibrium value would permit longer use of the vehicle before its solvent action became reduced.

Isopropyl alcohol first was used alone with the potassium chloride disc moved with Syntron action on a Buehler silk polishing cloth held over a lapped glass plate. This polishing action, again with 400 ml of alcohol, included a pressure of about 14 gr/cm² applied via a free-floating piston in a flanged polishing fixture. Rate of surface removal appeared to be about 600 Å/minute (see Figure 3), and the surface polish appeared to improve markedly with time. Scanning electron micrographs will be included in the next report. These rates are 200-600X slower than those reported recently in Honeywell's studies⁽⁸⁾ of acetic acid plus water combinations. The procedure will include a final etch step to eliminate all surface contaminant/abrasive.

Experiments during the third quarter will include a study of the action of both 0.05 micron size aluminum oxide (Linde B) in an isopropyl alcohol dispersion, as well as the use of isopropyl alcohol with soft polishing powders, including certain silicates and carbonates. This alcohol appears to have nearly optimum properties as an etch vehicle, with no apparent detrimental hydrolysis reaction problems. If the latter do become apparent, then the use of a trace of acetic acid (as per the Honeywell results) or an inorganic buffer combination will be utilized.



a. 170X Optical Quality, Center
Region (IIA₃ at 10 kv)

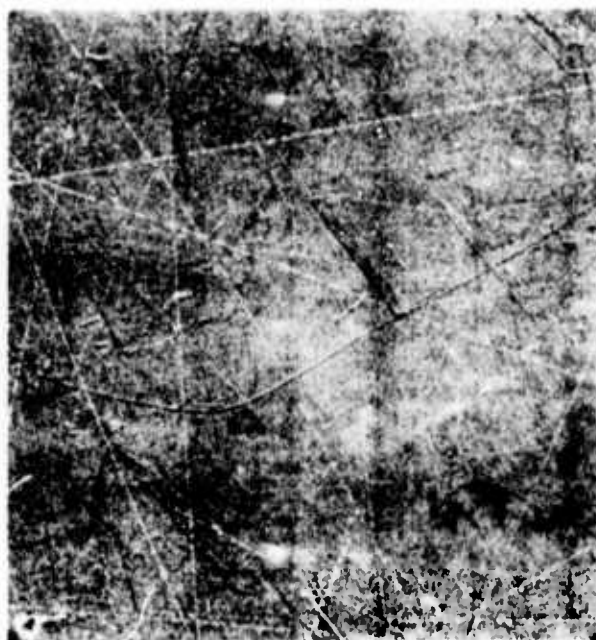


b. 150X Optical Quality, Edge
Region (IIC₁ at 10 kv)

Figure 2. Scanning Electron Micrographs of Harshaw Potassium
Chloride Windows Before Polishing Experiments



c. 3000X Optical Quality, Edge
Region (IID₁ at 10 kv)



d. 150X Laser Quality, Center
Region (IA₃ at 10 kv)

Figure 2. (Cont)

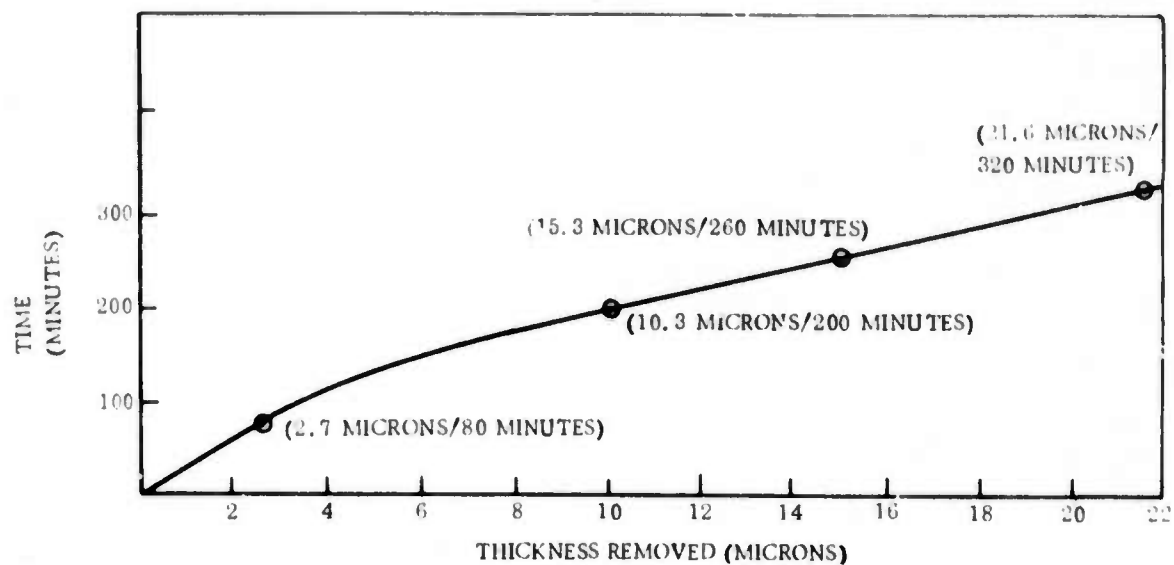


Figure 3. Removal of KCl by Nonabrasion Polishing in Isopropyl Alcohol Using a Syntro Polisher

Polishing utilizing low energy argon ion sputter etching techniques will be tried during the next quarter. Initial work with potassium chloride has indicated the sensitivity of this surface to ion damage.

3. SUMMARY

The purpose of this program is to conduct an integrated evaluation of techniques for surface finishing and protective coating of potassium chloride windows for high power IR laser applications. Thin film polymeric coatings are to be prepared by radio frequency glow discharge and ultraviolet techniques and their physical, chemical, and optical properties evaluated. The application of these materials as antireflective coatings will also be evaluated. Potassium chloride will be polished using chemical-mechanical and ion bombardment techniques and the finishes will be characterized before and after application of the protective coatings.

The design and assembly of the radio frequency glow discharge and ultraviolet photolysis equipment has been completed. The photolysis equipment has been modified to permit glow discharge cleaning of the potassium chloride windows prior to deposition of the polymeric coating. Deposition by a combination of glow discharge and ultraviolet photolysis is also possible through this modification.

Due to delays in receipt of several critical parts for the deposition equipment, only a few polymer deposition runs could be made during this quarter and the results were too preliminary to be definitive. The equipment is now completely assembled and final calibration of the instrumentation almost complete. Polymer deposition and evaluation will begin during the second week of the third quarter.

Polyhalogenated monomers were selected for evaluation based on their offering the best potential for meeting the optical, mechanical and chemical requirements of a protective coating for potassium chloride laser windows.

Surface properties of optically polished potassium chloride windows have been partially characterized by techniques which included scanning electron micrographs of surface scratches and defects. A new etch/polish process has been tested with several alcohols, and isopropyl alcohol has been shown to improve surface finish at about 600 Å/minute under Syntron vibration polishing. Further improvements of the process, including the use of soft polishing silicate and carbonate powders and hydrolysis-restrainers are planned for next quarter. The procedure will include a final etch step to eliminate all surface contaminant/abrasive.

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